Compositional analysis of additives containing Polyethylene by TGA

Introduction

The composition of polyethylene can be determined using different techniques and especially with the thermogravimetric analysis (TGA). The LABSYS evo TGA 1150 is very well adapted for performing the compositional analysis of polymers and plastics like polyethylene.

Experimental method

The compositional analysis is obtained from the thermogravimetric test that is used to determine the amounts of additives in a polyethylene compound. The test has to be performed firstly under nitrogen to decompose the organic matter and then under oxygen to burn the carbon content. The following experimental procedure was used in the presented example:

- Place a small piece (20 mg) of the polyethylene sample into the platinum crucible
- Apply a nitrogen flow rate (30 ml/min)
- Heat to from 30°C to 110°C at 5°C/min.
- Stabilize at 110°C during 30 minutes.
- Heat to from 110°C to 950°C at 10°C/min.
- Stabilize at 950°C during 30 minutes.
- Change the nitrogen gas to air (30ml/min) and stabilize at 950°C during 40 minutes.
Results

For the investigated polyethylene material which contained calcium carbonate and carbon black additives the following thermal events were observed:

- from 245°C to 500°C under nitrogen, decomposition of polyethylene
- from 500°C to 725°C under nitrogen, decarbonatation of CaCO₃ and formation of CaO
- At 950°C under air, combustion of the carbon black
- The remaining mass being the amount of CaO in the samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Mass loss 1 (%) 135°C to 500°C</th>
<th>Mass loss 2 (%) 500°C to 725°C</th>
<th>Mass loss 3 (%) 950°C</th>
<th>Remaining mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>PE + CaCO₃</td>
<td>97.77</td>
<td>0.98</td>
<td>N/A</td>
<td>1.25</td>
</tr>
<tr>
<td>PE + CaCO₃ + Carbon black 1</td>
<td>95.31</td>
<td>1.70</td>
<td>0.82</td>
<td>2.17</td>
</tr>
<tr>
<td>PE + CaCO₃ + Carbon black 2</td>
<td>94.95</td>
<td>1.82</td>
<td>0.92</td>
<td>2.31</td>
</tr>
</tbody>
</table>

Conclusion

The LABSYS evo TGA 1150 is very well adapted for the investigation of the decomposition of polymeric materials and especially the determination of the composition of polyethylene samples. For such a test, the Labsys TGA 1150 benefits from its flexibility in terms of temperature and atmosphere program for the definition of the different heating and cooling sequences, together with a very efficient and automated gas control device.

LABSYS evo TGA 1150 can also for instance be operated in accordance with a number of standard testing methods like:

- ASTM E1131 Standard Test Method for Compositional Analysis by Thermogravimetry, to determine accurately the amount of highly and medium volatiles, combustible and ash of compounds
- ASTM D6370 Standard Test Method for Rubber – Compositional Analysis by Thermogravimetry, to determine the organics and carbon black contents of rubbers
- ISO11358 – 1 and ISO11358 – 2 about Thermogravimetry (TG) of polymers